

Machine translation JP2001157581

(19)**Publication country**Japan Patent Office (JP)
(12)**Kind of official gazette**Publication of patent applications (A)
(11)**Publication No.**JP,2001-157581,A (P2001-157581A)
(43)**Date of Publication**June 12, Heisei 13 (2001.6.12)
(54)**Title of the Invention**A manufacturing method of a multienzyme granule
(51)**The 7th edition of International Patent Classification**

C12N 9/98

C11D 3/386

7/42

FI

C12N 9/98

C11D 3/386

7/42

Request for ExaminationUnrequested**The number of claims** 5**Mode of Application**OL**Number of Pages**6(21)**Application number**Japanese Patent Application No. 11-343349(22)**Filing date**December 2, Heisei 11 (1999.12.2)(71)**Applicant****Identification Number**000000918**Name**Kao Corp.**Address**1-14-10, Nihombashi Kayaba-cho, Chuo-ku, Tokyo(72)**Inventor(s)****Name**Raw ** ****Address**20, Higashi-Fukashiba, Kamisumachi, Kashima-gun, Ibaraki-ken Inside of the Kao Corp. research institute(72)**Inventor(s)****Name**Hayakawa Naoaki**Address**20, Higashi-Fukashiba, Kamisumachi, Kashima-gun, Ibaraki-ken Inside of the Kao Corp. research institute(72)**Inventor(s)****Name**Goro Ujita**Address**20, Higashi-Fukashiba, Kamisumachi, Kashima-gun, Ibaraki-ken Inside of the Kao Corp. research institute(74)**Attorney****Identification Number**100068700**Patent Attorney****Name**Ariga 3 happiness (outside trinomial)**Theme code (reference)**

4B050

4H003

F-term (reference)

4B050 CC07 CC08 DD02 JJ02 KK01 KK05 KK06 KK11 KK12

4H003 BA09 CA21 DA01 DA05 DA17 EA15 EA18 EA19 EA21 EA25 EB36 EB41 EC02 FA16

(57) Abstract

Means for Solution They are one sort or two sorts or more of enzyme powder containing two or more sorts of enzymes containing protease, Water-soluble particles are used as a core for enzyme powder which contains a stabilizing agent of this enzyme in parenchyma top homogeneity in enzyme powder containing an enzyme of a direction which is easy to be decomposed into this protease at least, A manufacturing method of a multienzyme granule corning using a water-soluble organic binder and covering with water solubility or water dispersibility coating subsequently.

Effect In this way, although the obtained multienzyme granule contains two or more sorts of enzymes containing protease, these enzymes are not deactivated and it is stability for a long period of time.

Claim(s)

Claim 1 They are one sort or two sorts or more of enzyme powder containing two or more sorts of enzymes containing protease, Water-soluble particles are used as a core for enzyme powder which contains a stabilizing agent of this enzyme in parenchyma top homogeneity in enzyme powder containing an enzyme of a direction which is easy to be decomposed into this protease at least, A manufacturing method of a multienzyme granule corning using a water-soluble organic binder and covering with water solubility or water dispersibility coating subsequently.

Claim 2 A manufacturing method of the multienzyme granule according to claim 1 which is that in which two or more sorts of enzymes containing protease contain two or more sorts of protease.

Claim 3 A manufacturing method of the multienzyme granule according to claim 1 whose stabilizing agents are one or more sorts of compounds chosen from a boron compound, inorganic acid alkaline earth metal salt, polyol, carboxylic acid, hydantoin, and urea.

Claim 4 A manufacturing method of a multienzyme granule of claim 1-3 whose stabilizing agent is a boron compound given in any 1 paragraph.

Claim 5 A manufacturing method of a multienzyme granule of claim 1-4 given in any 1 paragraph with which a stabilizing agent is contained one to 10% of the weight in enzyme powder.

Detailed Description of the Invention**0001**

Field of the Invention This invention relates to the manufacturing method of the multienzyme granule which blends two or more sorts of enzymes stably into the granulation of 1.

0002

Description of the Prior Art Protease is widely used for the detergent for garments in order to heighten a cleaning effect. In recent years, the detergent which blended two or more enzymes, such as different protease from cellulase, lipase, amylase, and the above other than protease, in order to heighten a detergency further is marketed.

0003 Although the method (namely, a granule is prepared for every enzyme) of usually blending one sort of enzymes with the granulation of 1 for blending two or more enzymes with a detergent is taken, if granule-ized equipment of 1 performs this, since we will be anxious about contamination of enzymes, etc., two or more granule-ized equipment is needed. Since it was **enzyme combination equipment / two or more** necessary in order to have blended two or more enzyme granules into the detergent, there was a problem that plant-and-equipment investment became great.

0004 Then, although the multienzyme granule which blended two or more enzymes into the granule of 1 is proposed, since other enzymes which are proteinic kinds will be disassembled by protein breakdown operation of protease when other enzymes are mixed with protease, the function may not be exhibited enough. The method (JP,61-168698,A) of dividing and blending with the nucleus part and husks portion of a granule, in order to avoid contact with protease in a granule, and other enzymes as a means to solve this, The enzyme granulation in which grain size differs is manufactured, and the method (Patent Publication Heisei No. 501840 **nine to**) of carrying out condensation enclosure of the large granule with a submicroscopic particulate, etc. are proposed. However, since these techniques need a complicated granulation method, granule

yield will fall.

0005

Problem(s) to be Solved by the Invention The purpose of this invention is to provide the manufacturing method of the multienzyme granule which blends stably efficiently two or more sorts of enzymes containing protease into the granule of 1.

0006

Means for Solving the Problem Then, in a concomitant use system of protease, this invention persons, and other enzymes, If blend a stabilizing agent of an enzyme of a direction which is easy to be decomposed into enzyme powder, water-soluble particles are used as a core, it corns using a specific binder and it covers, in spite of containing these enzymes in parenchyma top homogeneity in a granule of 1, It found out that a multienzyme granule by which disassembly of other enzymes by protease was prevented was obtained.

0007 Namely, this inventions are one sort or two sorts or more of enzyme powder containing two or more sorts of enzymes containing protease, Water-soluble particles are used as a core for enzyme powder which contains a stabilizing agent of this enzyme in parenchyma top homogeneity in enzyme powder containing an enzyme of a direction which is easy to be decomposed into this protease at least, It corns using a water-soluble organic binder, and a manufacturing method of a multienzyme granule covering with water solubility or water dispersibility coating subsequently is provided.

0008

Embodiment of the Invention As two or more sorts of enzymes (it may be hereafter called an ingredient (A)) containing the protease blended with the multienzyme granule of this invention, Combination with one or more sorts of enzymes (A²) chosen from protease (A¹), esterase, carbohydrase, lyase and the protease concerned, and different protease is mentioned. As an example of protease, pepsin, trypsin, chymotrypsin, collagenase, keratinases, elastase, subtilisin, papain, aminopeptidase, carboxypeptidase, etc. are mentioned. As an example of esterase, gastric lipase and blowout rare tic lipase, vegetable lipase, phospholipases, choline esterase, and phosphatase are mentioned. As carbohydrase, cellulase, maltase, saccharase, amylase, pectinase, alpha-, and beta-glycosidase etc. are mentioned. As lyase, carboxy lyase, aldehyde lyase, oxo acid lyase, hydro lyase, the lyase that acts on a polysaccharide, ammonia lyase, etc. are mentioned.

0009 Although (A¹) in these ingredients (A) and the compounding ratio in particular with (A²) are not limited, :(A¹) (A²) =1:1000-1000:1, especially 1:100 to 100:1 are preferred at a weight ratio.

0010 Although some stabilizing agents (it may be hereafter called an ingredient (B)) blended with the multienzyme granule of this invention are known as a stabilizing agent of various enzyme itself, it is not known at all that there is an effect which checks disassembly of other enzymes by protease like this invention. As an ingredient (B), one or more sorts chosen from a boron compound, inorganic acid alkaline earth metal salt, polyol, carboxylic acid, hydantoin, and urea are mentioned. Among these, a borax, boric acid, etc. are mentioned as a boron compound. As inorganic acid alkaline earth metal salt, the chloride of calcium or magnesium, sulfate, sulfite salt, a silicate, etc. are mentioned.

0011 Although a shook sirloin, mannitol, maltitol, sorbitol, trehalose, glycerin, propylene glycol, polysaccharide, etc. are mentioned as polyol, especially propylene glycol is preferred.

0012 As carboxylic acid, amino acid, fatty acid of the carbon numbers 1-20, dicarboxylic acid, hydroxycarboxylic acid, polysaccharide carboxylic acid, or its salt is more preferred. As amino acid or its salt, although a glycine, valine, leucine, aspartic acid, glutamic acid, arginine, phenylalanine, cysteic acid, serine, tryptophan, proline, lysine, methionine, or these salts are mentioned, Among these, valine, phenylalanine, histidine, proline, methionine, or these salts are more preferred. As other carboxylic acid or its salt, specifically, Carbonic acid, formic acid, acetic acid, propionic acid, adipic acid, succinic acid, maleic acid, Malonic acid, malic acid, citrate, alginic acid, or these salts are mentioned, among these calcium propionate, sodium formate, sodium acetate, magnesium acetate, calcium acetate, calcium carbonate, and especially the calcium alginate are preferred.

0013 As for especially these ingredients (B), it is preferred from a point of enzyme stability to blend two to 7% of the weight one to 10% of the weight into enzyme powder.

0014 in spite of containing protease and other enzymes in the ingredient (A) in this invention multienzyme granule -- an operation of an ingredient (B) -- being concerned -- others -- disassembly of an enzyme is prevented. For that purpose, the stabilizing agent (B) of the

enzyme (A²) needs to be included in parenchyma top homogeneity in the enzyme powder containing the enzyme (A²) which is easy to disassemble into protease (A¹) at least. The ingredient (A) and the ingredient (B) are contained in parenchyma top homogeneity in enzyme powder, or, specifically, the ingredient (A²) and the ingredient (B) should just be contained in parenchyma top homogeneity in enzyme powder.

0015As for uniformity of an enzyme and an ingredient (B), it is preferred to carry out for example, by adding and carrying out spray drying of the dehydrators, such as a salt cake, to enzyme solution an ingredient (B) and if needed. Especially the obtained enzyme powder has that preferred of **** for 10 - 40 weight sections nine to 100 weight section to nuclear particle 100 weight section.

0016The enzyme powder concerned is corned by using water-soluble particles as a core using a water-soluble organic binder, and, subsequently is covered with water solubility or water dispersibility coating.

0017Although it changes as water-soluble particles used as a core with enzymes to be used, particles, such as sodium chloride (salt), potassium chloride, a salt cake, sodium carbonate, and sugar, are mentioned, for example. As for the mean particle diameter of a nuclear particle, 0.2-1.2 mm is preferred. As for especially this nuclear particle, it is preferred to use 30 to 90% of the weight, into this invention multienzyme granule, so that it may become 40 to 60 % of the weight.

0018The polyethylene glycol whose (a) melting point is 35-80 ** as a water-soluble organic binder, and its derivative, The nonionic surface-active agent which is 80 ** is mentioned for the water soluble polymer chosen from the group which consists of a polyoxyethylene polyoxypropylene copolymer, (b) melting point, or the pour point, and one sort, such as 4000 or more polycarboxylic acid salt, or two sorts or more are mentioned for 35 - the (c) average molecular weight. As a water-soluble desirable organic binder, especially, As the polyethylene glycol of (a), and its derivative, a polyethylene glycol, Polyethylene-glycol sulfuric acid, a methoxy polyethylene glycol, etc. are mentioned, Polyoxyethylene alkyl ether etc. are mentioned as a nonionic surface-active agent of (b), and alkali metal salt, such as polyacrylic acid, an acrylic acid maleic acid copolymer, and polyacetal carboxylate, is mentioned as polycarboxylic acid salt of (c). Especially these water-soluble organic binders have that preferred of **** for 10 - 20 weight sections nine to 60 weight section to nuclear particle 100 weight section. As for the weight ratio of enzyme powder and a water-soluble organic binder, it is preferred to consider it as the range of the binders 0.2-2 to the enzyme powder 1.

0019In this invention, a powdered extender can be added further if needed. As an extender, talc, titanium oxide, zeolite, activated clay, kaolin, diatomite, bentonite, perlite, an acid earth, etc. are mentioned.

0020As a granulation means, dry granulation is preferred, and an extrusion granulation, a rolling granulation, a crack granulation, fluid bed granulation, spray granulation, a crushing granulation, etc. are mentioned as a granulation method. Among these, a rolling granulation method, especially a stirring rolling granulation method are preferred. As an example of stirring tumbling granulator, a Henschel mixer (Mitsui 3 Pond Chemical engineering machine), a high speed mixer (Fukae Industry), vertical granulator (Fuji Industry), etc. can be mentioned. These common appearances are having the vertical stirring shaft which attached agitating blades in the inside of the mixing chamber of a ** form. The REDIGE mixer (REDIGE) which is a granulator of the model which has a level stirring shaft can be used similarly.

0021As the water solubility or water dispersibility coating used for covering of the obtained granulation thing, Although not restricted in particular, a polyethylene glycol, polyacrylate, Polyvinyl alcohol, a polyvinyl pyrrolidone, a cellulosic, Water-soluble film formation polymer whose melting point of the derivative of starch etc. is 35-80 **; combination with protecting agents, such as polymer of these, the water solubility of talc, clay, titanium oxide, calcium carbonate, etc., a poorly soluble inorganic particle or an alkali metal silicate, and alkali metal carbonate, etc. is mentioned. As for coating, it is preferred 0.01 to 0.7 and to use especially at a rate of 0.05-0.6 to a granulation thing at a weight ratio.

0022As a coating method of a granulation thing, the method of a conventional method covering with devices, such as a fluidized bed granulator, a coating pan type granulator, and a stirring granulator, is mentioned.

0023Especially although the particle diameter in particular of the multienzyme granule obtained in this way is not restricted, 350-1500 micrometers is preferred 200-3000 micrometers as mean particle diameter.

0024this invention granule is useful as a combination ingredient of a detergent composition,

and the detergent composition which blended this can be used as detergents the object for garments, the object for tableware, for dwellings, etc.

0025

Example The salt cake was added in the solution of preparation enzyme powder 1 alkaline protease (the protease K, Bacillus sp.KSM-16 (FERM P11418) origin) of example 1(1) enzyme powder, it dried with the parallel flow type spray dryer, and the protease K content enzyme powder 1 was obtained. The content of the salt cake in the end of dried powder is 17%. The salt cake was added in the solution of enzyme powder 2 alkaline protease (the protease T, Bacillus sp.KSM-KP43 (FERM P-15863) origin), it dried with the parallel flow type spray dryer, and the protease T content enzyme powder 2 was obtained. The content of the salt cake in the end of dried powder is 10%.

The salt cake and the borax were added in the solution of enzyme powder 3 alkaline protease (protease T), it dried with the parallel flow type spray dryer, and the protease T content enzyme powder 3 was obtained. The salt cake in the end of dried powder and the content of a borax are 5% respectively.

(2), feeding altogether the raw material (a total of 3 kg except a coating agent) of the preparation following presentation of a multienzyme granule into a high speed mixer (Fukae Industry, FS-5J type), and pouring 70 ** warm water in a jacket. Stirring mixing was performed at agitator 360 r/min and chopper 900rpm, and contents were raised to 65 **. Then, the coating agent (total 90g) was supplied in the place which poured 40 ** warm water in the jacket and where contents were cooled to 50 **, and the multienzyme granules A and B were obtained. Not less than 350 micrometers 1000 micrometers or less of the recovery rate of the multienzyme granules A and B were not less than 98%.

0026

Table 1

For drawings please refer to the original document.

0027 The obtained multienzyme granules A and B were saved under 40 ** and the condition of 80% of relative humidity, and preservation stability was investigated. A result is shown in Table 2. It turns out that the multienzyme granule B which blended the borax from Table 2 is excellent in preservation stability.

0028

Table 2

For drawings please refer to the original document.

0029 The solution of the alkaline protease (protease K) which added 21 weight sections of preparation enzyme powder 4 salt cakes of example 2(1) enzyme powder to solid content 100 weight section of enzyme solution, Carrying out continuous mixing of the solution of the alkaline protease (protease T) which added 13 weight sections of salt cakes to solid content 100 weight section of enzyme solution, it dried with the parallel flow type spray dryer, and the enzyme powder 4 was obtained.

The solution of the alkaline protease (protease K) which added 21 weight sections of enzyme powder 5 salt cakes to solid content 100 weight section of enzyme solution, Carrying out continuous mixing of the solution of the alkaline protease (protease T) which carried out 4.1 weight-section addition of a salt cake and the borax 8.5 weight sections to solid content 100 weight section of enzyme solution respectively, it dried with the parallel flow type spray dryer, and the enzyme powder 5 was obtained.

(2) The raw material (a total of 3 kg except a coating agent) of the preparation following presentation of a multienzyme granule was altogether fed into the high speed mixer (Fukae Industry, FS-5J type), and the multienzyme granule was obtained like Example 1. Not less than 350 micrometers 1000 micrometers or less of the recovery rate of the multienzyme granules C and D were not less than 98%.

0030

Table 3

For drawings please refer to the original document.

0031The obtained multienzyme granules C and D were saved under 40 ** and the condition of 80% of relative humidity, and preservation stability was investigated. A result is shown in Table 4. It turns out that the multienzyme granule D which blended the borax from Table 4 is excellent in preservation stability.

0032

Table 4

For drawings please refer to the original document.

0033The multienzyme granule which blended two sorts of protease is prepared like example 3 Examples 1 and 2, When the residual activity is investigated, valine, phenylalanine, tryptophan, Proline, methionine, hydantoin, urea, sodium propionate, Calcium propionate, sodium formate, sodium acetate, magnesium acetate, The decomposition preventive effect of the enzyme by a magnesium chloride, calcium sulfate, calcium acetate, calcium carbonate, a calcium chloride, boric acid, the calcium alginate, and the protease that was extremely excellent in propylene glycol was accepted.

0034

Effect of the InventionAlthough the multienzyme granule obtained by this invention method contains two or more sorts of enzymes containing protease, these enzymes are not deactivated and it is stability for a long period of time.
